N91-21336

Containerless Processing Projects
Center for the Space Processing of Engineering Materials
Vanderbilt University, Nashville, Tennessee

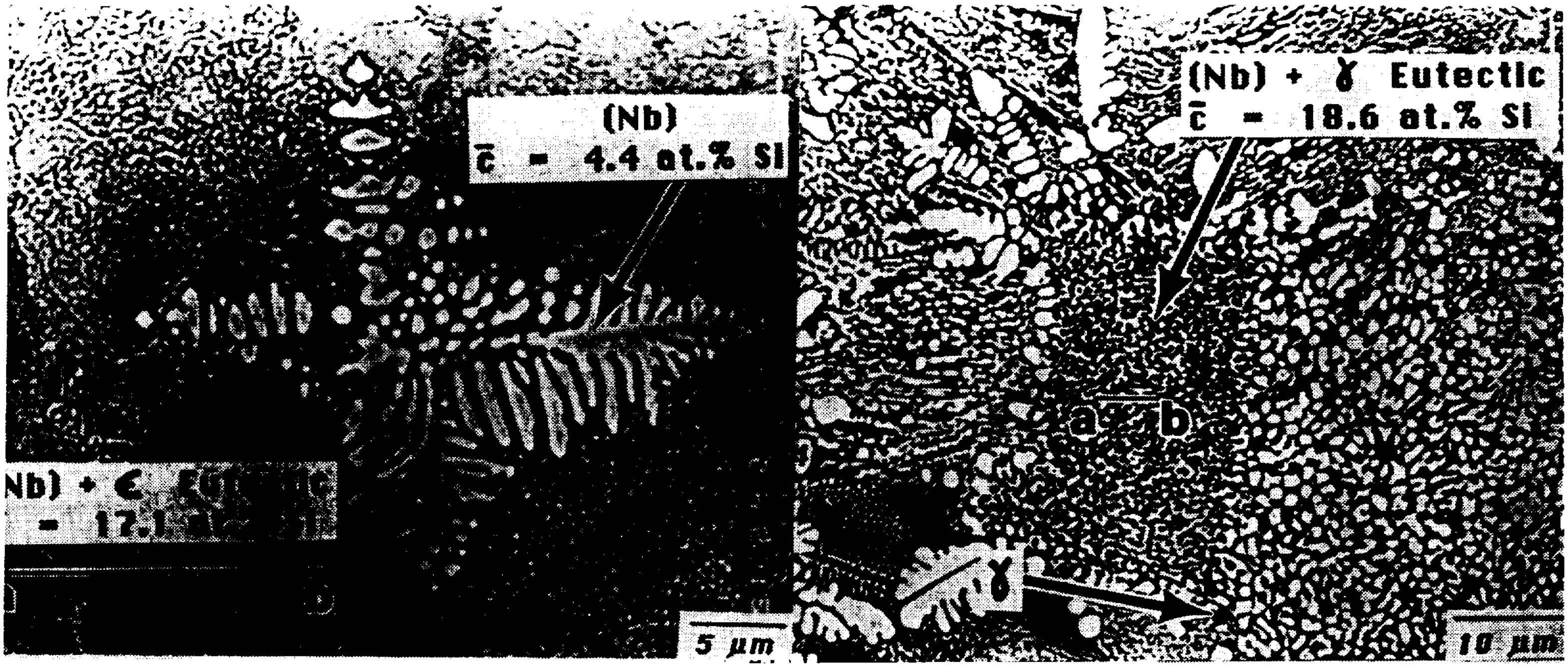
Alloy Investigations

- Ti-Al
- Ti-rare earth
- Nb-Ti
- Nb-Hf
- Nb-Si

Purification

- Nb
- Ir
- Ru

Development of Aerodynamic Levitation For Liquids



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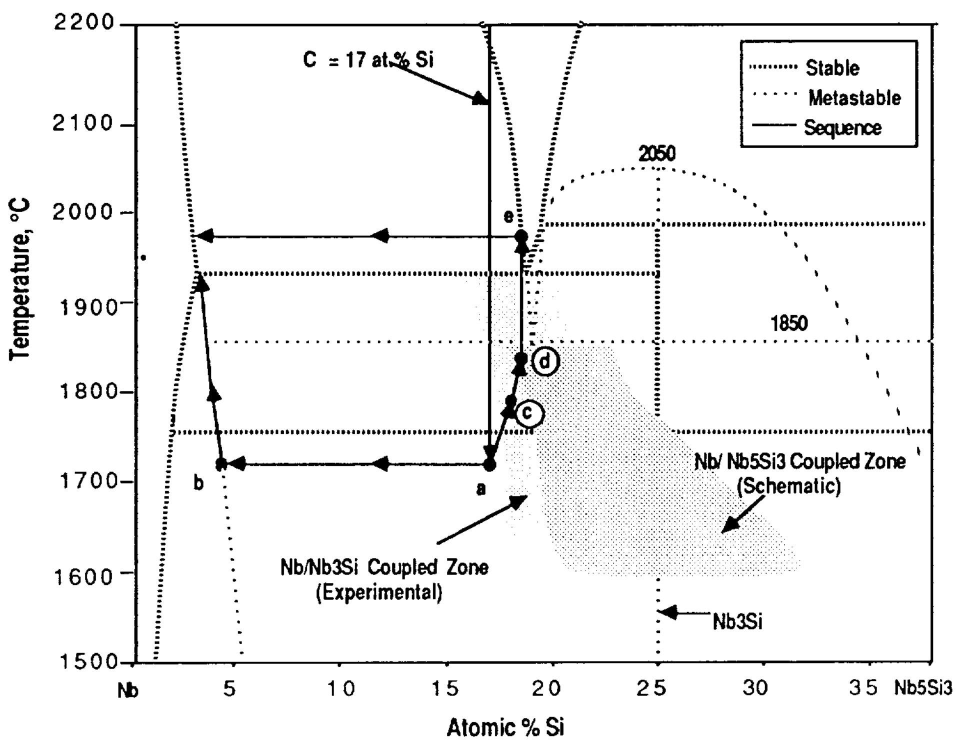


Advantages of Glass Coating
"Better reproducibility of achievement of high undercoolings"

1. Nearly independent of the surrounding gas atmosphere.
2. Nearly independent of cooling rate.
3. In ground-based work using viscous borosilicate glass coatings on nickel- and iron-based alloys, undercoolings above 300 K have been easily achieved.
4. Note that the undercoolings attained in ground-based experiments with glass coatings are equivalent to those attained under ultra-high vacuum without coatings.

Because:

(Crystalline inclusions and surface convection promote heterogeneous nucleation.)

1. Coatings prevent the formation of inclusions (e.g., oxides) due to reactions with the gas phase on the metal surface.
2. If such reactions occur, the softened glass will prevent the reaction products from forming crystalline inclusions.
3. Softened glass has a scavenging effect on inclusions within the metal specimen.
4. Glass coatings reduce surface convection due to lower surface tension and to increased viscous drag.

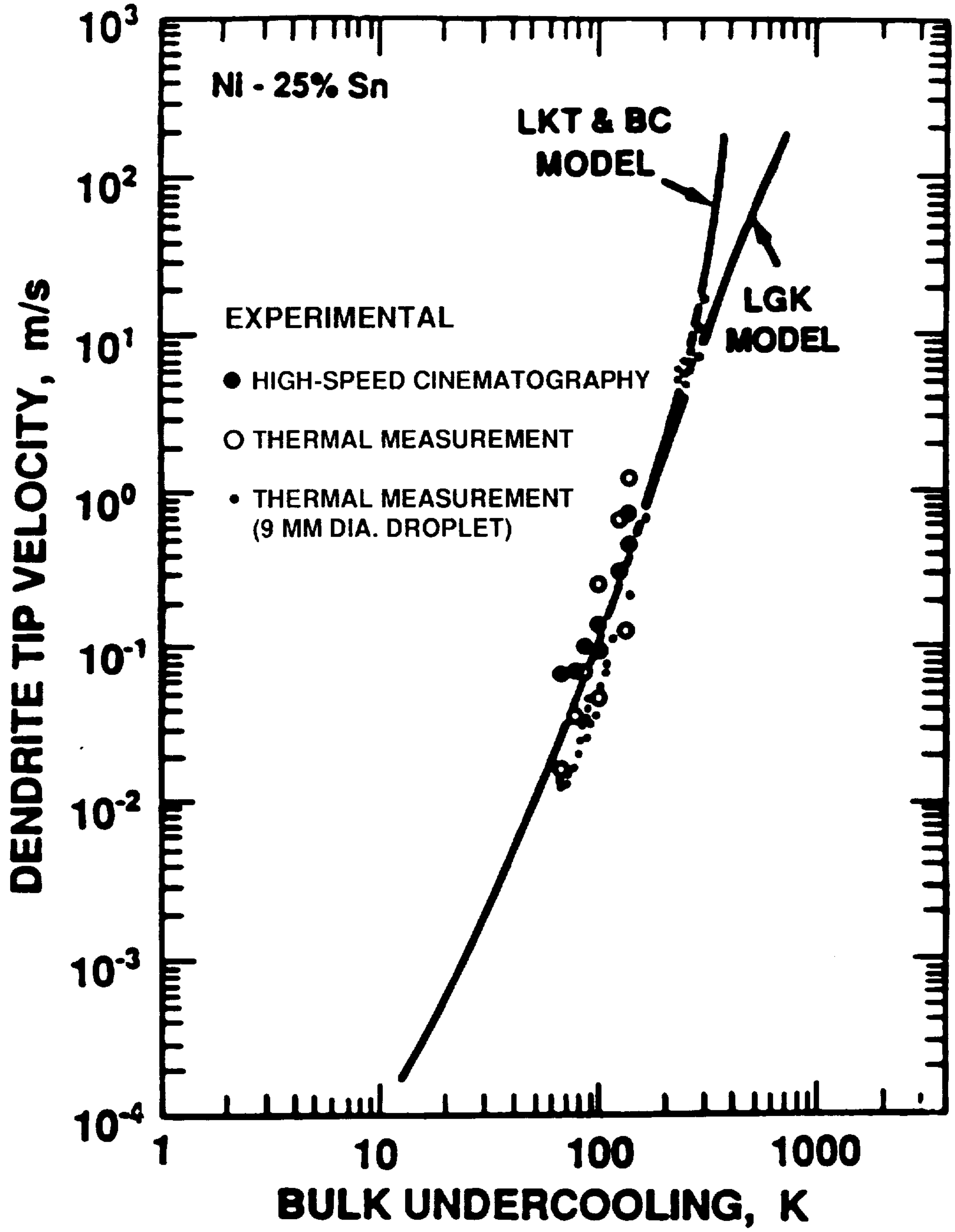


Figure 7. Dendrite tip velocity versus bulk undercooling for Ni-25 wt % Sn alloy. Experimental results and calculated curves based on the models developed by Lipton, Kurz, and Trivedi [35], Boettinger and Coriell [34] (LKT-BC), and by Lipton, Glicksman, and Kurz [33] (LGK).

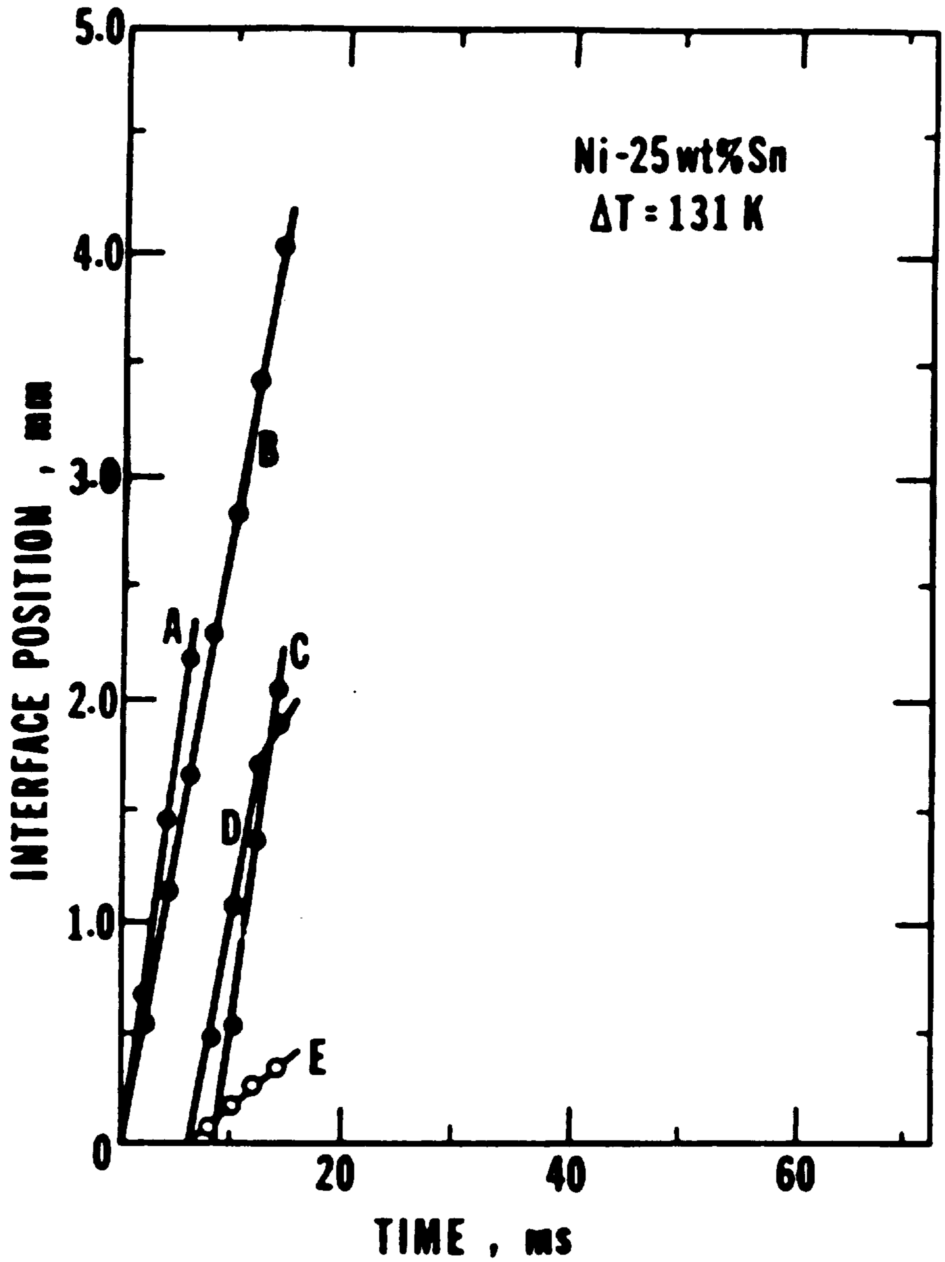


Figure 8. Plot of interface position as a function of time for the growth fronts in Fig. 1. The results for the four dendrites A, B, C, and D fall on straight lines, indicating that growth occurred at constant velocity (steady state).

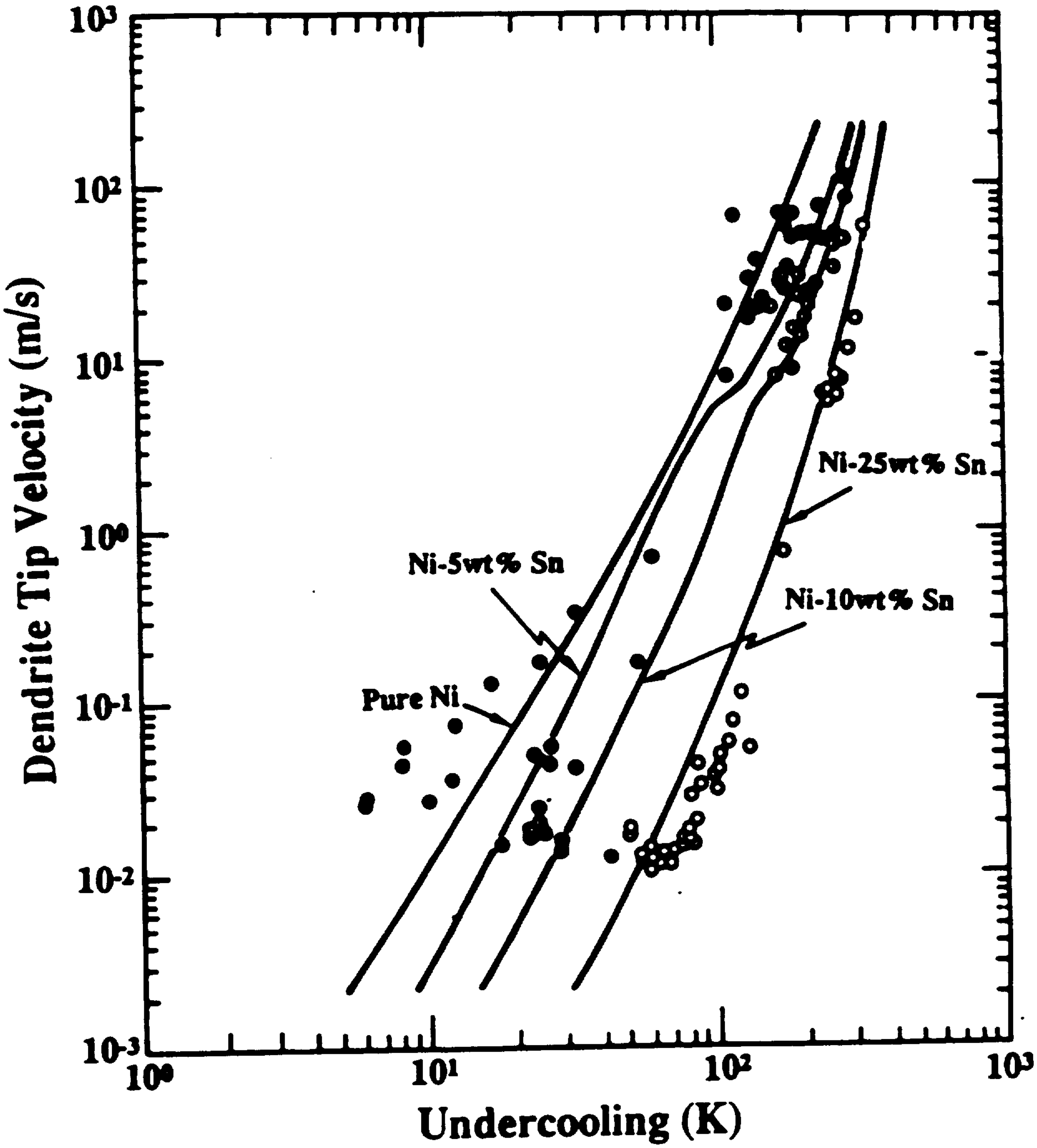


Figure 9. Log-log plot of dendrite tip velocity versus undercooling for pure Ni, Ni-5 wt% Sn, Ni-10 wt% Sn.

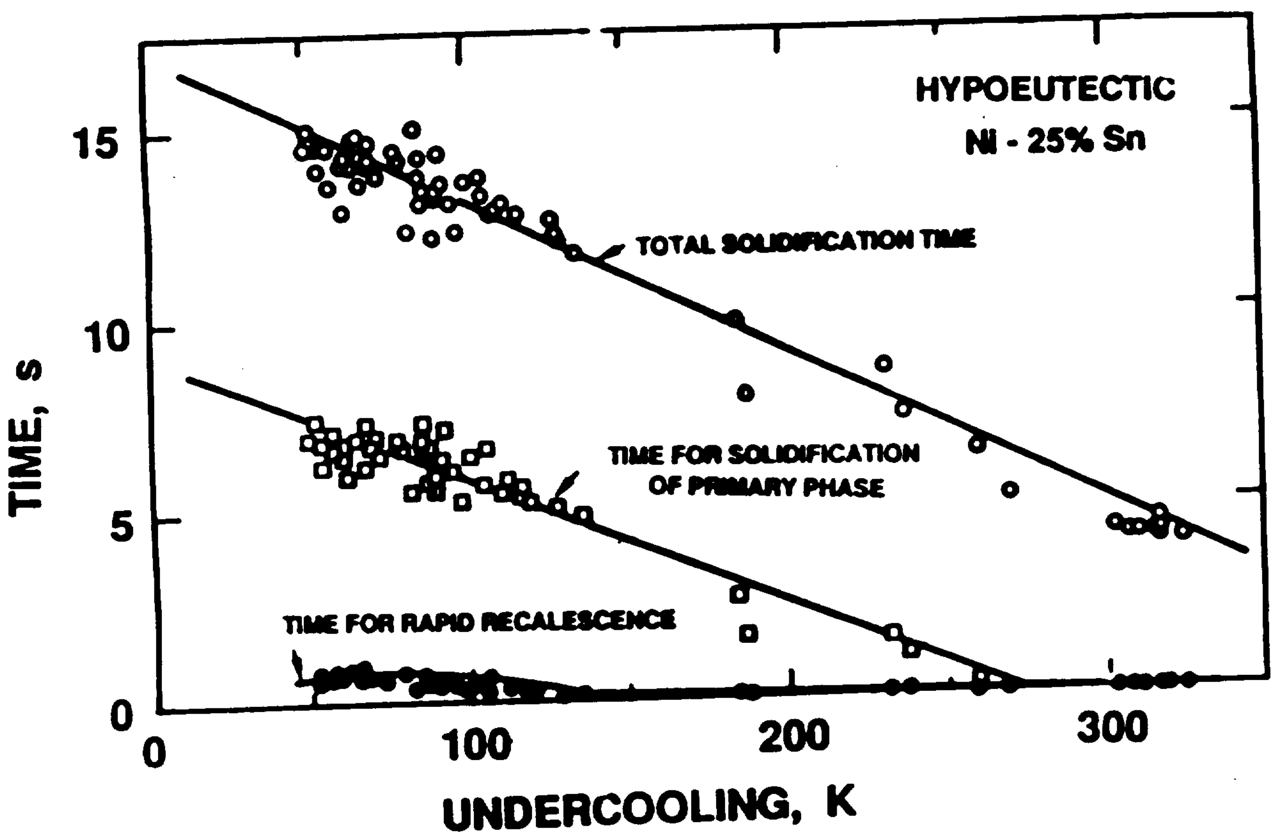


Figure 10. Plot of experimental results for solidification time, primary phase solidification time, and recalescence time versus initial undercoooling for Ni-25 wt% Sn alloy specimens.

PHYSICAL ACOUSTICS RESEARCH

- PRIMARY RESEARCH OBJECTIVE: Study fundamental acoustics of the DROP PHYSICS MODULE
- Develop Advanced Chamber Systems for Acoustic and Sample Characterization
- Study Sample Interaction With Acoustic Field
- Study Shaping of Liquid Drops by Acoustic Forces
- Study the acoustic torque as a function of sample position and shape
- Study Levitated Sample Stability and the Effects of Feedback Systems

PHYSICAL ACOUSTICS RESEARCH

- PRIMARY RESEARCH OBJECTIVE:
  Study fundamental acoustics of the DROP PHYSICS MODULE

- Develop Advanced Chamber Systems for Acoustic and Sample Characterization
  - Study optimization of chamber acoustic power
  - Study chamber - sound source coupling
  - Study different chamber dimensions
  - Study different sound source orientations

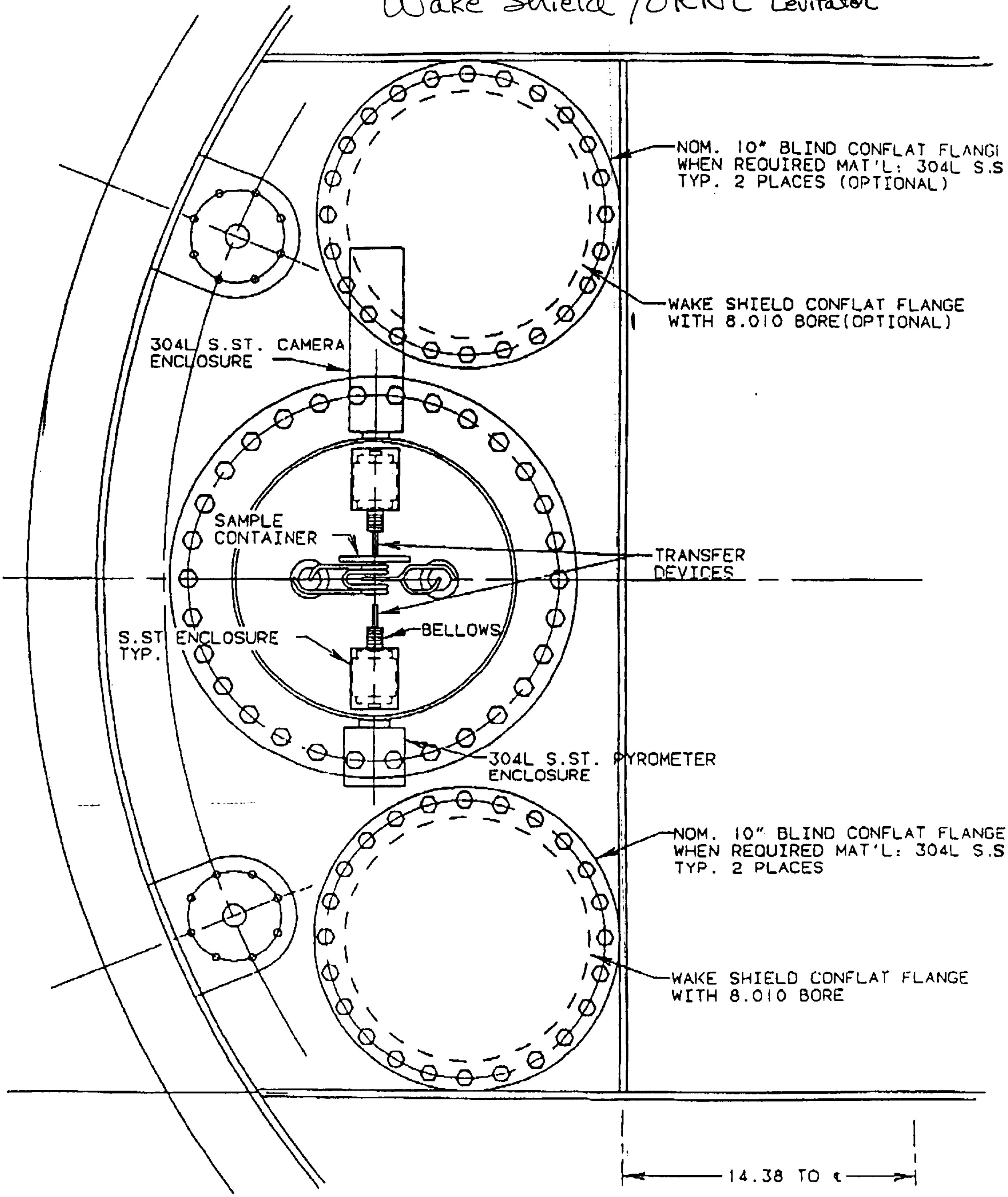
- Study Sample Interaction With Acoustic Field
  - Study sample scattering effects on higher harmonics as a function of:
    - Sample size
    - Sample shape
    - Sample position

- Study Shaping of Liquid Drops by Acoustic Forces
  - Study sample shape as a function of sample size, position, and acoustic field intensity
  - Study acoustic force and pressure profiles for various normal mode resonances

- Study the acoustic torque as a function of sample position and shape

- Study Levitated Sample Stability and the Effects of Feedback Systems
  - Intrinsic instabilities caused by: frequency, amplitude, temperature drifts
  - Extrinsic instabilities caused by: frequency, amplitude, phase modulations of driver signals

Wake Shield /ORNL Levitator



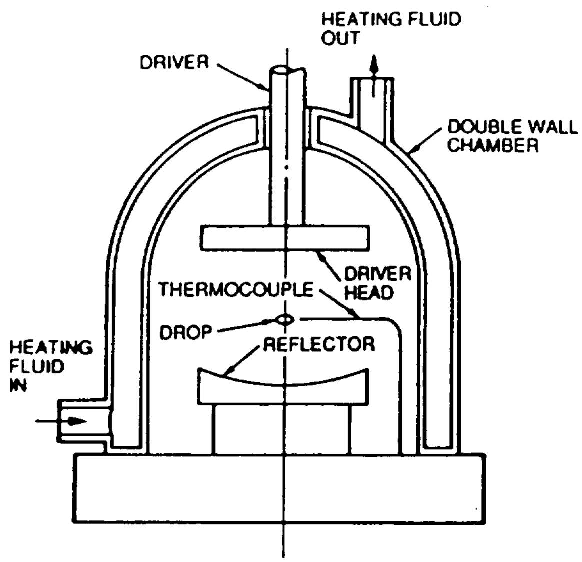


Fig. 1. The heart of a single axis acoustic levitation system.

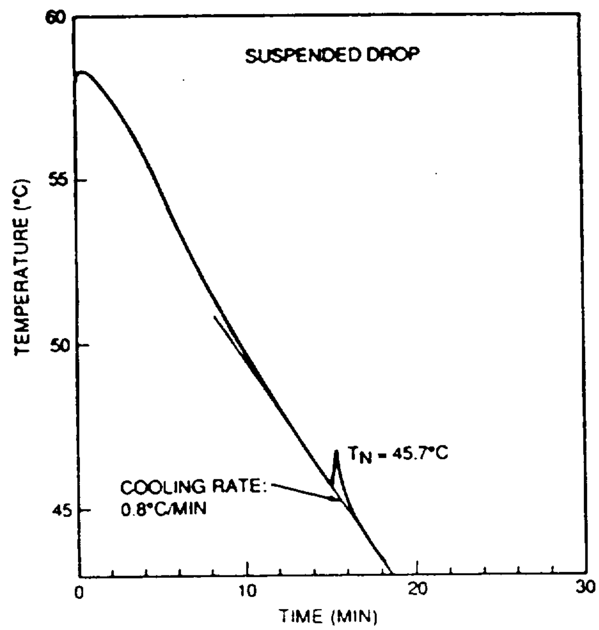


Fig. 3. A cooling curve of a levitated succinonitrile drop.

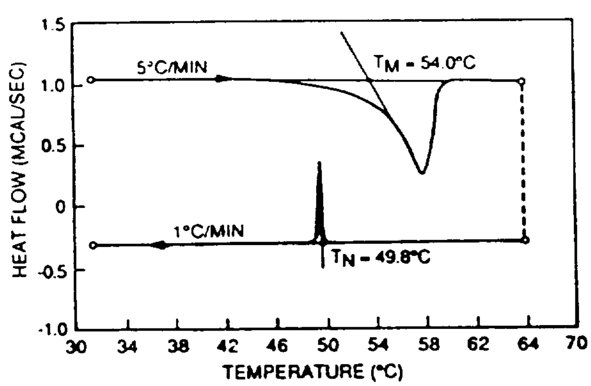


Fig. 2. A DSC thermogram showing melting and solidification of succinonitrile.

INVESTIGATORS ON TEMPUS FOR IML-2 MISSION

Effects on Nucleation by Containerless Processing in Low Gravity - R.J. Bayuzick and W.H. Hofmeister, Vanderbilt University; M.B. Robinson, George C. Marshall Space Flight Center, NASA

Viscosity and Surface Tension of Undercooled Melts - I. Egry, DLR Institute of Space Simulation

Alloy Undercooling Experiments - M.C. Flemings, Massachusetts Institute of Technology; H.D. Brody, University of Pittsburgh

Non-Equilibrium Solidification of Largely Undercooled Melts - D.M. Herlach, DLR Institute of Space Simulation

Metallic Glass Research in Space - W.L. Johnson and H.J. Fecht, California Institute of Technology; M.C. Lee, NASA Headquarters

Measurement of the Viscosity of the Undercooled Melts Under the Conditions of Microgravity - J. Szekely, Massachusetts Institute of Technology

EXPERIMENTAL OUTLINE

- Niobium and zirconium -- pure and ultrahigh pure

- Five processing conditions:

  - high vacuum electromagnetic levitation on earth

  - ultrahigh vacuum electromagnetic levitation on earth

  - high vacuum drop tube

  - ultrahigh vacuum drop tube

  - ultrahigh vacuum electromagnetic processing in LEO

- 100 undercooling experiments planned for each case to obtain histograms of nucleation frequency as a function of temperature

  - maximum undercooling

  - most probable nucleation temperature

  - dispersion in nucleation temperature

PROTOCOL

Insert individual specimen into position for heating

Heat specimen until fully molten and soak for about 30 seconds

At about 2570°C for Nb
At about 1955°C for Zr

Cool specimen until solidification occurs - cooling rate to be determined - most likely to be the natural cooling rate with no power input

Monitor thermal history and brightness history

Monitor vacuum/environment history

Monitor power history - both sets of coils

Repeat for approximately 100 cycles

Place specimen in individual container to retain identity

Repeat entire process for remaining three specimens

Entire set consists of two Nb specimens and two Zr specimens - a total of four specimens

Alloy Undercooling Experiment

OBJECTIVES:

To study the rapid solidification after undercooling of melted metal spheres levitated in microgravity and the resulting microstructures.

To obtain a semi-quantitative understanding of the effect of gravity on the containerless solidification of small diameter metal alloy spheres.

APPROACH:

Melt glass-coated spheres of nickel-tin and iron-nickel alloys in low-gravity by levitation melting.

Cause undercooling by heat withdrawal after power cutoff.

Obtain thermal history (cooling and recalescence) by pyrometry.

Observe solidification behavior in situ during recalescence by cinematography.

Perform metallographic studies on processed specimens.

Perform ground-based experiments for comparison with microgravity experiments.

Preliminary Science Requirements

TEMPUS is nearly ideal for MIT
alloy undercooling experiments.

Specific Concerns

Temperature Measurement
0.4-2 μm due to glass coating
Separate output for each detector
Careful calibration

Sample Rotation and Stability
Minimum possible for Fe-B, Fe-P

Solidification Front Recording
Video: Maximum Possible for Fe-Ni and Ni-Sn
500 fields per second > required for Fe-B, Fe-P

Sample Capture
Quenching, e.g. liquid metal.

IMMEDIATE OBJECTIVES

Solidification of highly undercooled levitation melted alloys.

Solidification of highly undercooled alloys in microgravity.

ULTIMATE OBJECTIVES

Achieve hypercooling.

Rapid solidification of bulk material.

Fundamental understanding of rapid solidification.

ULTIMATE OBJECTIVES

HYPERCOOLING

$$\Delta T = \Delta H / C_p + (T_L - T_S)$$

ΔT (K)	Alloy
500	Ni - 25 wt% Sn
450	Ni - 1 wt% Sn
375	Fe - 25 wt% Ni
440	Ni - 10 wt% Cu

Practical Implications

If hypercooling can be achieved in microgravity:

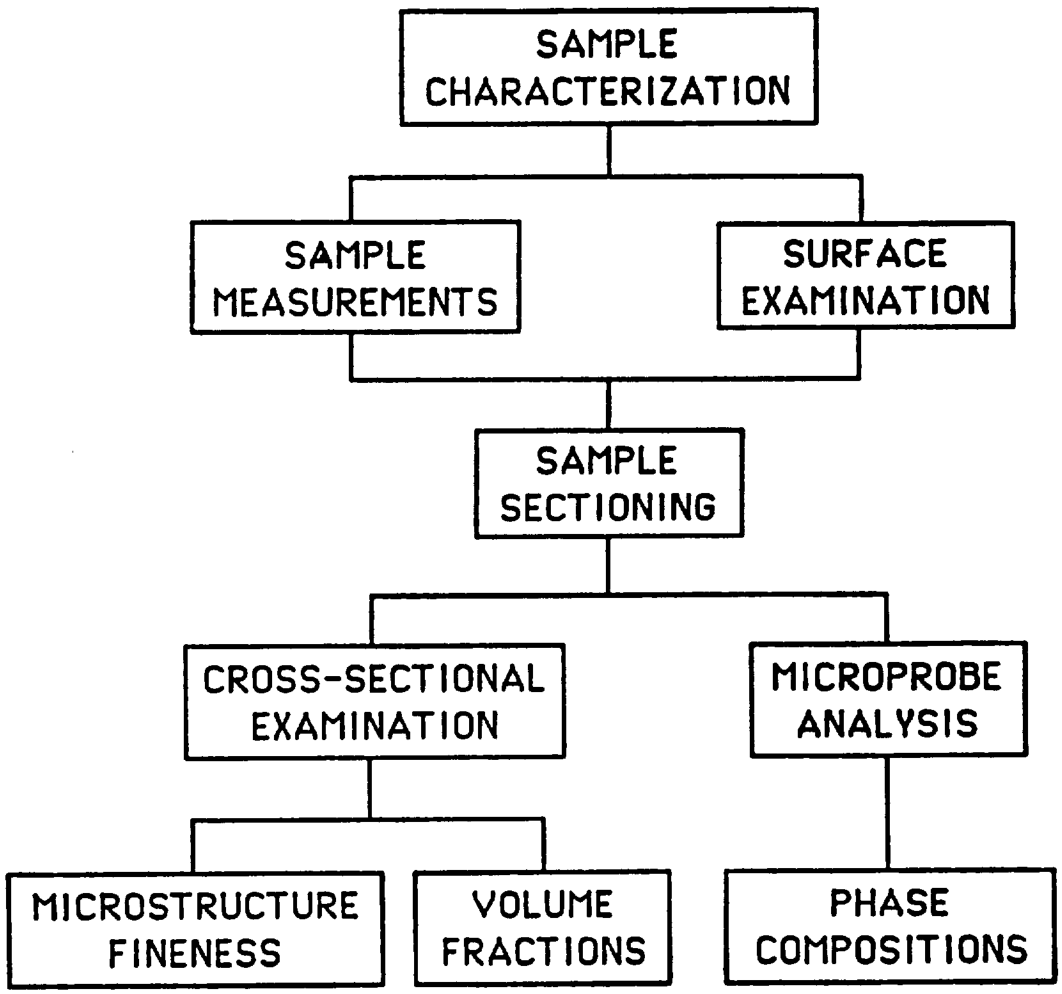
No upper size limit on fully homogeneous and metastable RSP materials as exists at 1 G.

Topics of scientific interest:

Growth rates achieved are thermally controlled (solute trapping).

Morphology of the solidification front (absolute stability).

Convection effects in rapid solidification.


SAMPLE CHARACTERIZATION
SAMPLE MEASUREMENTS
SURFACE EXAMINATION
SAMPLE SECTIONING
CROSS-SECTIONAL EXAMINATION
MICROPROBE ANALYSIS
MICROSTRUCTURE FINENESS
VOLUME FRACTIONS
PHASE COMPOSITIONS

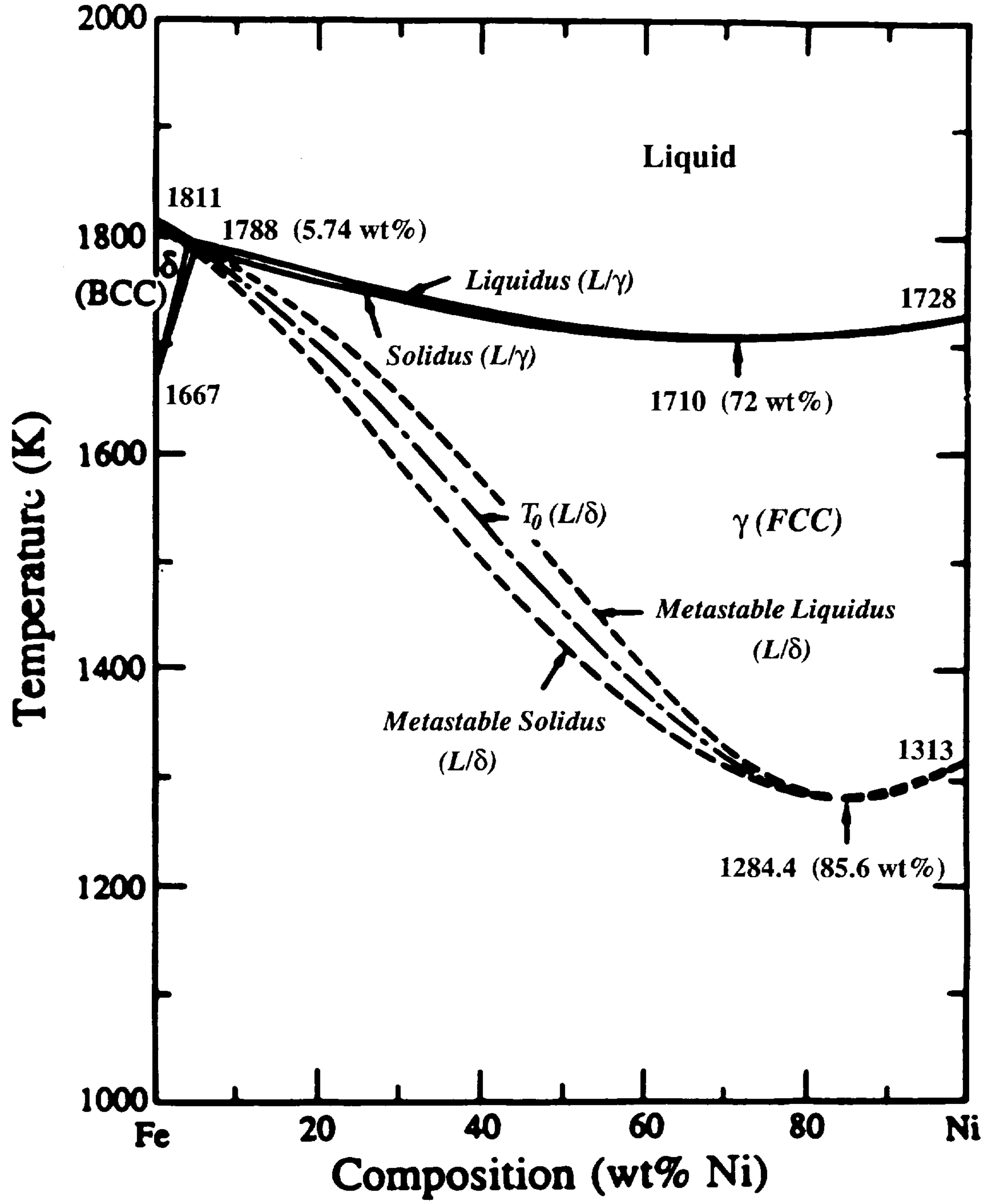
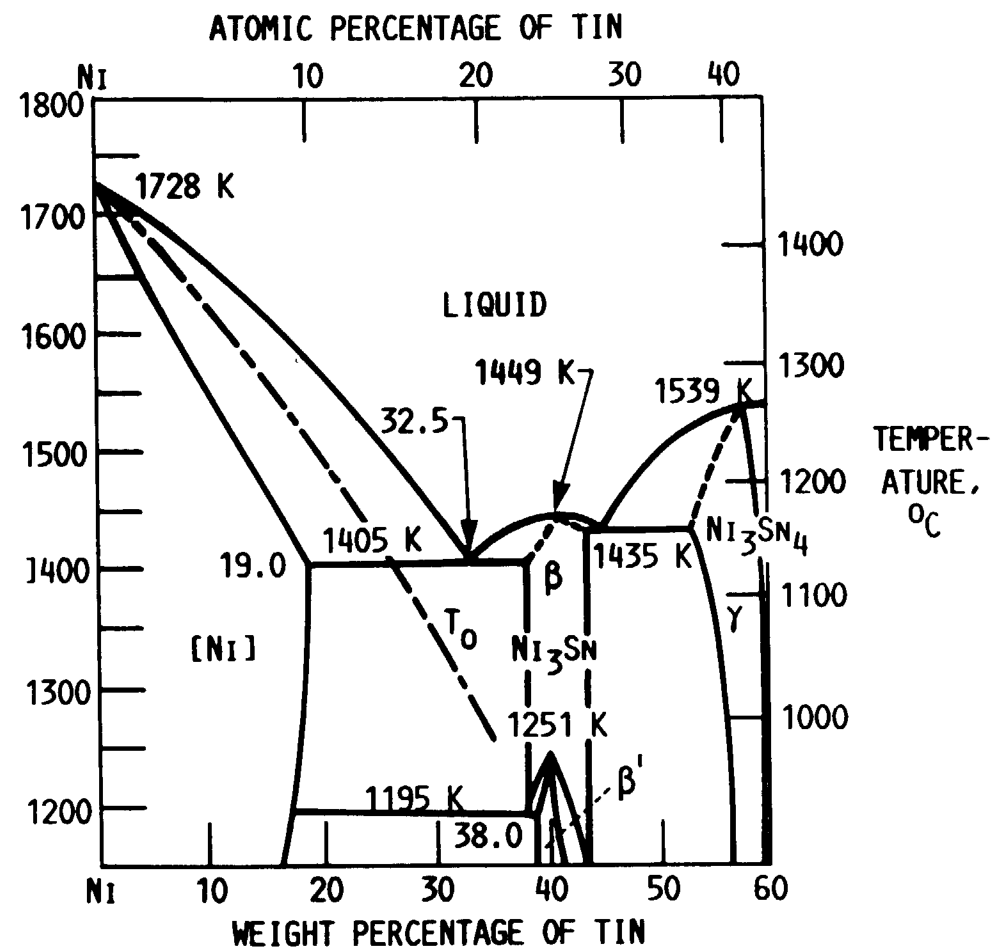


Figure 10. The Fe-Ni phase diagram, with the metastable extensions of the liquidus, solidus, and "T_0" lines for the BCC δ phase.

EML - ALLOY UNDERCOOLING EXPERIMENT

MATERIAL

Ni-32.5 wt% Sn EUTECTIC M.P.
1405 K SPHERE 8.7 mm DIAMETER
WITH 0.1 mm THICK GLASS COATING



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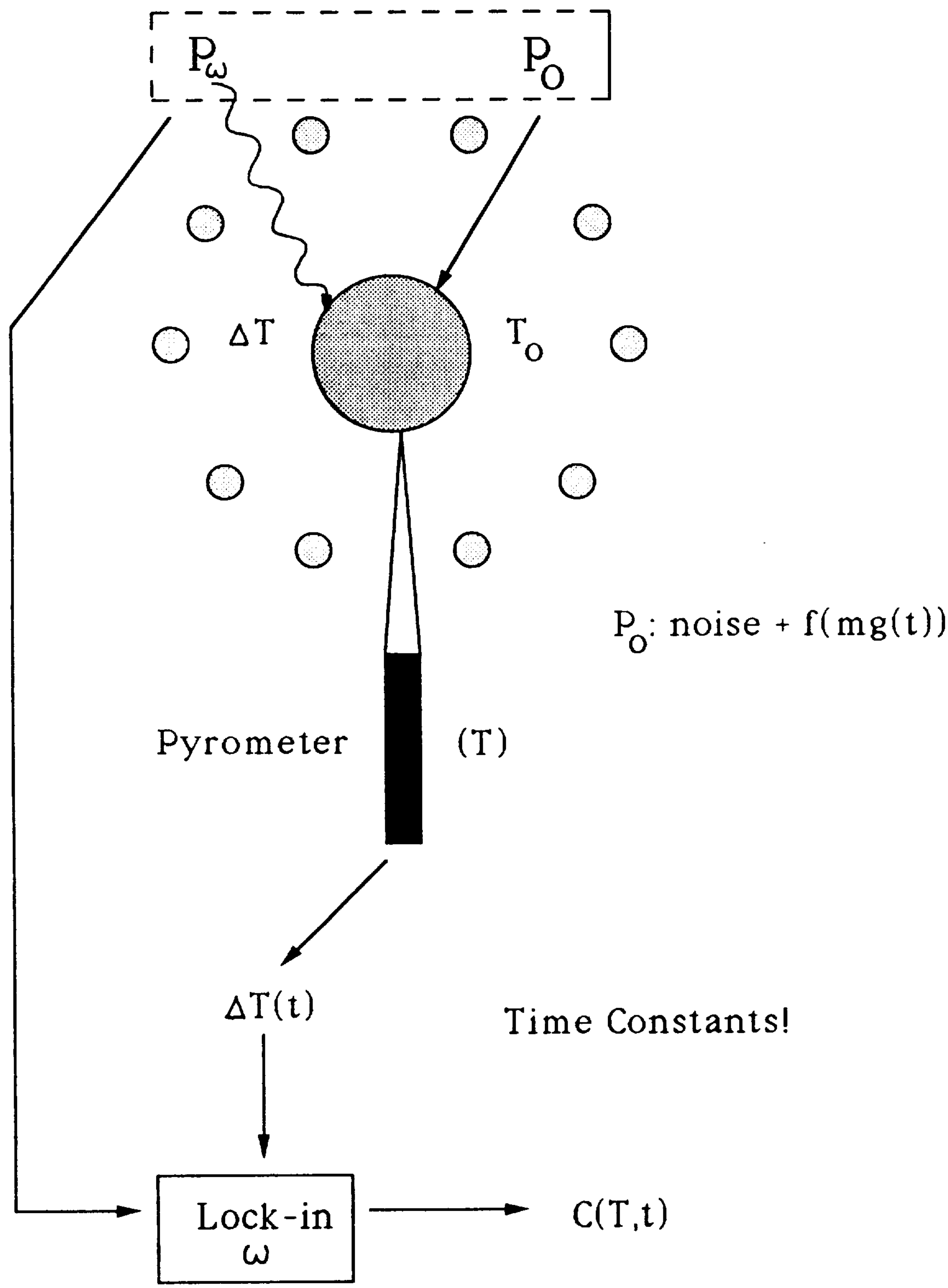
P_ω
P_o
ΔT
T_o
P_o: noise + f(mg(t))
Pyrometer
(T)
$\Delta T(t)$
Time Constants!
Lock-in
ω
C(T,t)